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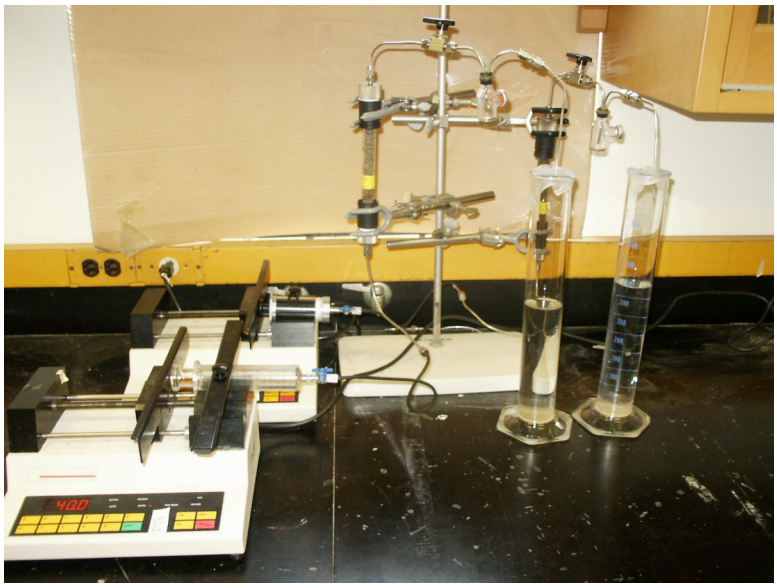
APPENDIX G

TREATABILITY TEST DATA REPORT

SAUGET AREA 1

Treatability Tests

Sauget and Cahokia, Illinois



January 6, 2006

Groundwater Services, Inc.
2211 Norfolk, Suite 1000, Houston, Texas 77098

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1. INTRODUCTION

As part of the DNAPL Characterization and Remediation Study for Sauget Area 1, a number of bench-scale treatability tests were developed and conducted. These tests were designated Task 6 in the DNAPL Work Plan. The results from these tests are intended to provide design information for evaluating the applicability and potential effectiveness of the technologies selected.

The technologies chosen for evaluation were 1) surfactant-enhanced solubilization, 2) dissolution, 3) thermal treatment, and 4) chemical oxidation. Assessments of surfactant-enhanced solubilization and dissolution were conducted on a bench-scale using samples from Sauget Area 1. As an alternative to conducting a detailed thermal treatability bench-scale test, recovered DNAPL was characterized by generating a boiling point curve (ASTM D86 Distillation Test). An evaluation of chemical oxidation was performed using results from previous bench-scale testing conducted for a nearby site.

The following information is included for each technology:

- Objectives
- Approach
- Experimental Procedures
- Results and Discussion

All tests were conducted at and in cooperation with the laboratories in the Environmental Engineering department at Rice University in Houston, Texas. Sample analyses were performed by Severn Trent Laboratories (STL) in Savannah, Georgia.

Design data generated during the treatability tests were used to provide information for the evaluation and comparative analysis of control measures for the site.

2. TEST 1—SURFACTANT-ENHANCED SOLUBILIZATION

2.1 Treatability Study Objectives:

Surfactant-enhanced solubilization was considered as a potential aid to accelerate remediation of the source area. Enhancement is achieved by the addition of a chemical surfactant with the goal of altering the physical-chemical environment of the contaminant such that the effective aqueous solubility is increased. This transfer of the contaminant from the oil phase to the aqueous phase means the compound is more amenable to flushing or other remedial efforts.

Objectives of the treatability study included:

- Determine the applicability of two different types of surfactants for enhancing solubility
- Provide design data for mass of surfactant required per mass of DNAPL present and correlate to cost per volume of soil treated

2.2 Technical Approach

Surfactant flushing (with or without cosolvent) has been developed as an aggressive remediation technology for DNAPL contamination in the subsurface (Yin and Allen, 1999). The feasibility of this technology is based on the interaction between the surfactant and the contaminants in relation to the media in which they are present, typically water. Surfactants have the ability to alter the interfacial properties of fluids, with the end result of enhancing the amount of mass that can be present in an aqueous phase at equilibrium (Edwards et al., 1991).

Surfactants are classified as compounds that contain both a hydrophobic moiety (typically a long chain hydrocarbon) and a hydrophilic moiety. Differences in the molecular composition of the latter group are used to classify surfactants as ionic, non-ionic, or cationic. Because of the amphipathic nature of surfactants, they are soluble in water yet form aggregates (called micelles) with the hydrophobic ends grouped centrally towards each other. At a surfactant-specific critical micelle concentration (CMC), all subsequent additions of surfactant will associate with these micelles. The hydrophobic centers of the micelles provide a favorable location for association with hydrophobic organic contaminants, and the hydrophilic tails allow these aggregates to be part of the aqueous phase. The effective solubility of a contaminant generally increases linearly beyond the CMC because of continued micelle formation (Simpkin et al., 1999).

Similarly, surfactant amendments are intended to enhance mobilization *in situ* by lowering the interfacial tension between DNAPL and the surrounding aqueous phase. The interaction between each molecule of contaminant and the surfactant acts to dissipate capillary forces. In dealing with a non-aqueous phase contaminant that has a higher relative density than water, this type of mobilization may be undesirable because it can lead to downward movement and pooling of the contaminant. However, it is generally difficult to separate the effects of enhanced solubilization and mobilization. The treatability tests described here did not attempt to account for mobilization effects because the assays involved only liquid phases and were soil-free.

The goal of a surfactant flood is to move compounds that are sparingly soluble and relatively immobile (due to capillary forces and/or sorption to soil) into the aqueous phase (Abriola et al., 1995). This is accomplished at concentrations greater than the CMC, a concentration that ranges between 10 and 10,000 mg/L for typical surfactants. In field applications, even higher concentrations are often used to counter uneven distribution and non-equilibrium mass transfer (Simpkin et al., 1999). In these treatability tests, two surfactants (Aerosol MA-80I and Tween 80)

were used at two different concentrations that were greater than their respective CMCs. The concentrations were similar to those used in field demonstrations of this technology. Aerosol MA-80I (sodium dihexyl sulfosuccinate) is an anionic surfactant used successfully for demonstrating enhanced DNAPL remediation at Hill Air Force Base in Logan, Utah in 1997 (Simpkins et al., 1999). Tween 80 (polysorbate 80) is a non-ionic surfactant that has been studied extensively due to its extensive use as a food additive and does not appear to uniformly inhibit biological activity (including dechlorination of chlorinated benzenes) (McGuire and Hughes, 2003). Both compounds are FDA-approved and General Recognized as Safe.

For the tests described in this report, surfactants were added to solutions containing DNAPL of known composition that was recovered from the Sauget Area 1 site. The composition of this DNAPL is detailed in the data report, but contains significant mole fractions of various chlorinated benzenes and ethenes.

Surfactant test data were obtained using an experimental procedure that was somewhat modified from the procedure outlined in the "Work Plan for DNAPL Characterization and Remediation Study" dated April 1, 2004. The Work Plan called for each surfactant to be evaluated by: i) adding NAPL to water in a beaker or closed jar and gently mixing; ii) sampling and analyzing the aqueous phase for VOCs and SVOCs; iii) adding surfactant slowly and mixing until the CMC is observed visually; iv) taking selected measurements of NAPL-water interfacial tension as surfactant concentration is increased to the CMC; and v) analyzing post CMC aqueous phase for VOCs and SVOCs.

The critical micelle concentration (CMC) represents the minimum amount of surfactant that must be present in a solution to begin to increase the solubility of compounds affected by the surfactant. At concentrations above the CMC, solubility of affected compounds tends to increase in a linear relationship. Increasing the solubility of non-aqueous phase components was a goal of the surfactant treatability test, but it should be noted that surfactant floods in subsurface applications are rarely performed at the CMC. Rather, the surfactant concentration is typically much higher (> 0.5% v/v) to take advantage of the relatively linear relationship between the component solubility and the surfactant concentration. In addition, the higher surfactant concentration used in field applications provides a safety factor in terms of overcoming limitations in hydraulic control or multi-phase contact. For these reasons, it was more appropriate to assess the solubilization enhancement that could be obtained at higher levels of each surfactant.

Data were obtained using an experimental procedure that was somewhat modified from the procedure outlined in the Work Plan in order to maximize the potential for successfully achieving enhanced solubilization. The test was conducted at equilibrium conditions, thereby negating mass transfer limitations. The test was conducted using concentrations of surfactant that exceeded the CMC, so that the response in dissolved concentration enhancements for each mg/L of surfactant added was at its maximum. Conducting the treatability test using the original method would have decreased the potential degree of enhancement observed, and thus would have lessened any observable solubilization effects.

2.3 Experimental Methods and Materials

Methods. The test involved two different surfactants (Aerosol MA-80I and Tween 80) added at two different concentrations. In addition, the concentration of VOCs and SVOCs before surfactant was added were assessed in duplicate reactors. Bottles used for the pre-surfactant addition analysis of VOCs and SVOCs were not used further because of losses that may have occurred during sampling. Instead, new bottles were set up using the same initial conditions and amended with appropriate amounts of surfactant. Therefore, 6 reactors were set-up and analyzed:

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Reactor ID	Surfactant Type	Surfactant Concentration (% by weight)
STT-1A	---	---
STT-1B	---	---
STT-2A	Tween 80	0.5
STT-2B	Tween 80	1.0
STT-3A	Aerosol MA-80I	1.0
STT-3B	Aerosol MA-80I	2.0

All test reactors were 4-L bottles containing 10% (by weight) DNAPL recovered from well BR-I at Sauget Area 1 (Figure 2.1). Total liquid volume was 2.5 L, with the majority of the volume composed of deionized water buffered with 1000 mg/L HCO_3^- to maintain a pH of 6.7. All reactors were capped during the majority of the experiment (with the exception of the sampling phase). DNAPL (25 mL) was transferred by pipette to the bottom of reactors filled with 2.5 L of DI water and allowed to equilibrate over the course of 2 days. No active mixing was employed because the sampling protocol necessitated separation of DNAPL and aqueous phases. Even with solely passive mixing, there was some coating of the glass surfaces with an oil phase. Following the equilibration phase, a set of two bottles were sampled for initial VOCs and SVOCs. Two of the remaining 4 bottles were amended with a 100 mL aliquot of a concentrated solution of Tween 80, and the final 2 bottle were amended with a 100 mL aliquot of a concentrated solution of Aerosol MA-80I. These pre-mixed concentrated solutions consisted of aliquots of the corresponding surfactant dissolved in DI water and allowed to equilibrate over the course of 2 days. Each was formulated such that 100 mL additions of each would result in the desired mass ratios in the 4-L reactor bottles, specifically 0.5 or 1.0% w/w of Tween 80, and 1.0 or 2.0% w/w of Aerosol MA-80I. These final concentrations were selected because they were 1) similar to those used in field surfactant floods, and 2) above the manufacturers' provided CMCs. The surfactant solutions were added to the bottom of each reactor, with care taken to minimize disturbance of the DNAPL. Passive mixing was employed to ensure mass transfer in the multi-phase system over the course of the next 4 days. After this period, all reactors were again sampled for VOCs and SVOCs. Duplicate samples were collected from the two reactors that received no surfactant amendment.

Analysis. Samples for analysis of volatile organic compounds (VOCs) were collected in 40 mL vials with TFE. Vials were filled with no headspace, and hydrochloric acid was used as a preservative. Samples were transferred to the vials via pipette to minimize disturbance of the DNAPL layer at the bottom of each reactor. Duplicate samples were collected and analyzed using EPA Method 8260. Samples for analysis of semi-volatile organic compounds (SVOCs) were collected in 1 L amber bottles with screw-top caps and TFE. Samples were transferred to the vials via pipette to minimize disturbance of the DNAPL layer at the bottom of each reactor. Samples were collected and analyzed using EPA Method 8270. Sample blanks were included for both analytical methods. All sample analyses were conducted by Severn Trent Laboratories (STL) in Savannah, Georgia. Collected samples were shipped overnight in coolers on ice, and all bottles were stored at 4ºC until ready to ship.

Chemicals. DNAPL recovered from the site (estimated 100 mL volume) and analyzed by STL was used in the test. Aerosol MA-80I was provided by Cytec Industries (Willow Island, WV) in liquid form at 80% active by weight. The manufacturer's listed CMC was 7100 mg/L. Tween 80 was purchased from Sigma-Aldrich (St. Louis, MO) in liquid form at 100% active by weight. The manufacturer's listed CMC was 13 mg/L.

2.4 Results and Discussion

Data generated during the surfactant treatability test are located in Table 2.1. The concentration of VOCs and SVOCs before the addition of surfactant was assessed in two 4 L reactors (STT-1-Start and STT-2-Start). The goal was to determine effective solubilities of various compounds at equilibrium, and to establish baseline concentrations to assess the impact of surfactant amendments. Effective solubilities for each compound can be estimated for each compound based on the mole fraction of each in the recovered DNAPL that was used in the treatability test. For example, 1,2,4-trichlorobenzene was present at a mole fraction of 0.20, and the expected concentration at equilibrium would be 9.9 mg/L (or 0.20 multiplied by the pure phase solubility of 48.8 mg/L). As shown in Table 2.1, the measured concentration of 1,2,4-trichlorobenzene was 2.2 mg/L at the onset of the experiment, which is significantly below the expected concentration. When the same methodology is applied to other compounds previously identified in the recovered DNAPL, a similar disparity between the expected effective solubility and the measured concentration was noted for naphthalene, benzene, tetrachloroethene, ethylbenzene, chlorobenzene, and xylene. In general, the measured concentrations for this set of compounds ranged from 10 to 30% of the calculated solubilities. In addition, a number of compounds not identified in the recovered DNAPL were detected in the test reactors. These included toluene, nitrobenzene, chloroaniline, acetone, methyl ethyl ketone (MEK), carbazole, fluorene, phenol, n-nitrosodiphenylamine, and a number of chlorinated phenolic compounds.

In the unamended reactors, the total SVOC concentration averaged 3.0 mg/L and the total VOC concentration averaged 0.12 mg/L.

Samples were collected from the reactors containing surfactant following the 4 day equilibration period. Aqueous samples from the Aerosol MA-80I-amended reactors crystallized during the extraction and analysis steps for SVOCs. Therefore, the only data available for Aerosol MA-80I are VOC concentrations.

The data following the addition of each surfactant at two different levels is shown in Figure 2.2. Total VOC concentration was determined by summing the masses of individual volatile compounds, and a similar procedure was followed for SVOCs for Tween 80. Surfactant concentrations above the CMC represent practical levels for study because COC effective solubilities should increase above this threshold. However, for Tween 80, the total concentration of COCs (calculated as VOC+SVOC for Tween 80) did not increase following the addition of surfactant. For a 0.5% w/w solution of Tween 80, the total COC concentration decreased from 2.95 mg/L to 2.59 mg/L, and dropped farther to 1.89 mg/L following the addition of surfactant at 1% w/w. Trends for each compound class (SVOC and VOC), as well as trends for individual compounds, were similar. For Aerosol MA-80I, the total VOC concentration increased from 0.12 mg/L to 2.4 mg/L after the addition of 1.0% w/w, but VOCs dropped to 1.9 mg/L after increasing the surfactant concentration to 2.0% w/w. Acetone and methyl isobutyl ketone (MIBK) were the compounds responsible for the majority of the increase in concentration relative to the unamended control. No consistent enhancement in solubilization was not noted for any of the other compounds of interest. For example, an increase in the ethylbenzene concentration at 1.0 % w/w of Aerosol MA-80I was followed a concentration decrease when more surfactant was added. Similarly, many compounds detected at 0.5 % w/w of Tween 80 either decreased in concentration or were undetectable at the higher surfactant concentration (1.0% w/w).

The results from this treatability test suggest that surfactant-enhanced solubilization is not an appropriate technology selection for the Sauget Area 1 site. Because increases in concentration following surfactant addition were not observed, no estimates can be made of the mass of surfactant needed to remove the constituents present at the site. While it is possible that surfactant amendments may have a more measurable impact on solubilization *in situ*, there is

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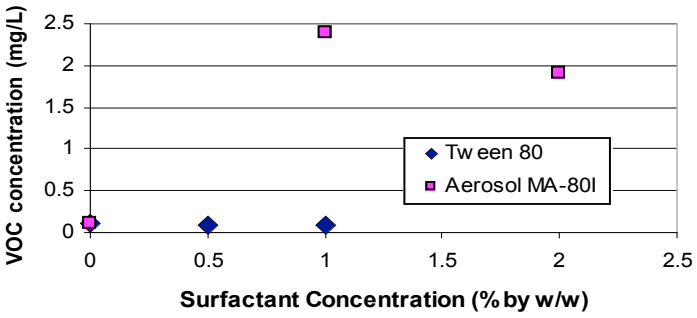
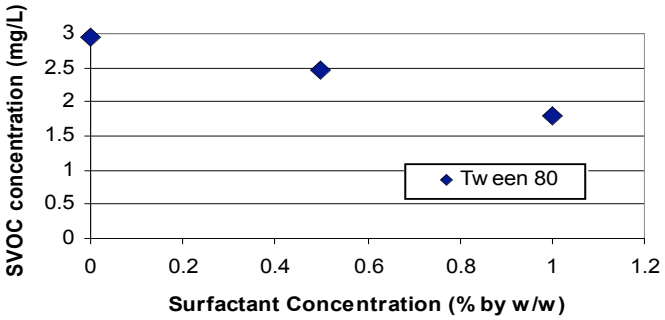
little indication that the compound profile is amenable to this technology. Given that the bulk unit cost of Tween 80 is roughly $1/lb of surfactant, the uncertainty associated with the effectiveness of the tested surfactants has to potential to unfavorably multiply the potential supply costs of surfactant solubilization.



(a)



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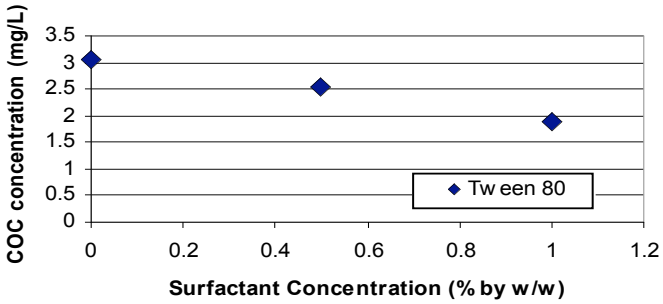

(c)

Figure 2.1. Surfactant-enhanced solubilization test using DNAPL recovered from Sauget Area 1. (a) VOC concentration (b) SVOC concentration, and (c) COC concentration for each surfactant. The data points displayed for unamended reactors (0% w/w) represent baseline concentrations. No SVOC data is available for Aerosol MA-80I reactors.

Table 2.1
Total VOC and SVOC Concentrations: Surfactant Treatability Test

DNAPL Characterization Study
Sauget Area 1, Sauget, Illinois

	SAMPLE ID:	STT-1A	STT-1B	STT-2A	STT-2B	STT-3A	STT-3B
	SAMPLE DATE:	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04
Analyte	**CAS No.**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**
Volatile Organic Compounds by EPA Method 8260							
1,1,1-Trichloroethane	71-55-6	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
1,1,2,2-Tetrachloroethane	79-34-5	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
1,1,2-Trichloroethane	79-00-5	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
1,1-Dichloroethane	75-34-3	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
1,1-Dichloroethene	75-35-4	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
1,2-Dichloroethane	107-06-2	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
1,2-Dichloropropane	78-87-5	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
2-Butanone (MEK)	78-93-3	**0.0028J**	<0.05	<1	<0.5	<1	<1
2-Hexanone	591-78-6	<0.02	<0.05	<1	<0.5	**0.14J**	<1
4-Methyl-2-pentanone (MIBK)	108-10-1	<0.02	<0.05	<1	<0.5	**0.58J**	**1**
Acetone	67-64-1	**0.053**	<0.12	<2.5	<1.2	**1.4J**	**0.56J**
Benzene	71-43-2	**0.014**	**0.017**	<0.1	<0.05	<0.1	<0.1
Bromodichloromethane	75-27-4	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Bromoform	75-25-2	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Bromomethane	74-83-9	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Carbon Disulfide	75-15-0	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Carbon Tetrachloride	56-23-5	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Chlorobenzene	108-90-7	**0.035**	**0.049**	**0.053J**	**0.056**	**0.068J**	<0.1
Chloroethane (ethyl chloride)	75-00-3	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Chloroform	67-66-3	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Chloromethane	74-87-3	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
cis-1,2-Dichloroethene	156-59-2	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
cis-1,3-Dichloropropene	10061-01-5	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Dibromochloromethane	124-48-1	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Dichloromethane	75-09-2	**0.021**	**0.031**	**0.038J**	**0.036J**	**0.049J**	**0.037J**
Ethyl benzene	100-41-4	<0.01	<0.025	<0.5	<0.25	<0.5	<0.5
Styrene	100-42-5	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Tetrachloroethene	127-18-4	**0.002**	**0.0033J**	<0.1	<0.05	<0.1	<0.1
Toluene	108-88-3	**0.0048**	**0.0064**	<0.1	<0.05	<0.1	<0.1
trans-1,2-Dichloroethene	156-60-5	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
trans-1,3-Dichloropropene	10061-02-6	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Trichloroethene	79-01-6	**0.00092J**	0.005	<0.1	<0.05	<0.1	<0.1
Vinyl Chloride	75-01-4	<0.002	<0.005	<0.1	<0.05	<0.1	<0.1
Xylenes,Total	1330-20-7	**0.0025J**	**0.003J**	<0.2	<0.1	**0.15J**	**0.26**
Total VOCs		**0.14**	**0.11**	**0.091**	**0.092**	**2.4**	**1.9**

Notes:
1. All samples were analyzed by Severn Trent Laboratories (STL), Savannah, Georgia.
2. Detected analytes are presented in bold type.
3. J = Analyted detected below quantitation limits. < = Analyte not detected at the specified reporting limit.

Table 2.1
Total VOC and SVOC Concentrations: Surfactant Treatability Test

DNAPL Characterization Study
Sauget Area 1, Sauget, Illinois

	SAMPLE ID:	STT-1A	STT-1B	STT-2A	STT-2B
	SAMPLE DATE:	12/2/04	12/2/04	12/2/04	12/2/04
Analyte	**CAS No.**	**mg/l**	**mg/l**	**mg/l**	**mg/l**
Semivolatile Organic Compounds by EPA Method 8270					
1,2,4-Trichlorobenzene	120-82-1	**2.2**	**2.2**	**1.8**	**1.3**
1,2-Dichlorobenzene	95-50-1	**0.073**	**0.089**	**0.087J**	**0.062J**
1,3-Dichlorobenzene	541-73-1	<0.01	<0.01	<0.1	<0.1
1,4-Dichlorobenzene	106-46-7	**0.32**	**0.38**	**0.3**	**0.22**
2,4,5-Trichlorophenol	95-95-4	**0.0071J**	**0.0077J**	**0.034J**	0.1
2,4,6-Trichlorophenol	88-06-2	**0.061**	**0.063**	**0.029J**	**0.049J**
2,4-Dichlorophenol	120-83-2	**0.061**	**0.075**	<0.1	**0.047J**
2,4-Dimethylphenol	105-67-9	<0.01	<0.01	<0.1	<0.1
2,4-Dinitrophenol	51-28-5	<0.05	<0.05	<0.5	<0.5
2,4-Dinitrotoluene	121-14-2	<0.01	<0.01	<0.1	<0.1
2,6-Dinitrotoluene	606-20-2	<0.01	<0.01	<0.1	<0.1
2-Chloronaphthalene	91-58-7	<0.01	<0.01	<0.1	<0.1
2-Chlorophenol	95-57-8	<0.01	<0.01	<0.1	<0.1
2-Methylnaphthalene	91-57-6	<0.01	<0.01	<0.1	<0.1
2-Methylphenol (o-Cresol)	95-48-7	<0.01	<0.01	<0.1	<0.1
2-Nitroaniline	88-74-4	<0.05	<0.05	<0.5	<0.5
2-Nitrophenol	88-75-5	<0.01	<0.01	<0.1	<0.1
3,3'-Dichlorobenzidine	91-94-1	<0.02	<0.02	<0.2	<0.2
3-Methylphenol/4-Methylpheno	106-44-5	<0.01	<0.01	<0.1	<0.1
3-Nitroaniline	99-09-2	<0.05	<0.05	<0.5	<0.5
4,6-Dinitro-2-methylphenol	534-52-1	<0.05	<0.05	<0.5	<0.5
4-Bromophenylphenyl ether	101-55-3	<0.01	<0.01	<0.1	<0.1
4-Chloro-3-methylphenol	59-50-7	<0.01	<0.01	<0.1	<0.1
4-Chloroaniline	106-47-8	**0.021**	**0.03**	<0.2	<0.2
4-Chlorophenylphenyl ether	7005-72-3	<0.01	<0.01	<0.1	<0.1
4-Nitroaniline	100-01-6	<0.05	<0.05	<0.5	<0.5
4-Nitrophenol	100-02-7	<0.05	<0.05	<0.5	<0.5
Acenaphthene	83-32-9	<0.01	<0.01	<0.1	<0.1
Acenaphthylene	208-96-8	<0.01	<0.01	<0.1	<0.1
Anthracene	120-12-7	<0.01	<0.01	<0.1	<0.1

continued

Notes:
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Table 2.1
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DNAPL Characterization Study
Sauget Area 1, Sauget, Illinois

	SAMPLE ID:	STT-1A	STT-1B	STT-2A	STT-2B
	SAMPLE DATE:	12/2/04	12/2/04	12/2/04	12/2/04
Analyte	**CAS No.**	**mg/l**	**mg/l**	**mg/l**	**mg/l**
Semivolatile Organic Compounds by EPA Method 8270					
Benzo(a)anthracene	56-55-3	<0.01	<0.01	**0.013J**	<0.1
Benzo(a)pyrene	50-32-8	<0.01	<0.01	<0.1	<0.1
Benzo(b)fluoranthene	205-99-2	<0.01	<0.01	<0.1	<0.1
Benzo(g,h,i)perylene	191-24-2	<0.01	<0.01	<0.1	<0.1
Benzo(k)fluoranthene	207-08-9	<0.01	<0.01	<0.1	<0.1
bis(2-Chloroethoxy)methane	111-91-1	<0.01	<0.01	<0.1	<0.1
bis(2-Chloroethyl)ether	111-44-4	<0.01	<0.01	<0.1	<0.1
bis(2-Ethylhexyl)phthalate	117-81-7	<0.01	**0.003**	<0.1	<0.1
Butylbenzylphthalate	85-68-7	<0.01	<0.01	<0.1	**0.012J**
Carbazole	86-74-8	**0.0017J**	**0.0017J**	<0.1	0.1
Chrysene	218-01-9	0.01	0.01	**0.012J**	0.1
Dibenzo(a,h)anthracene	53-70-3	<0.01	<0.01	<0.1	<0.1
Dibenzofuran	132-64-9	<0.01	<0.01	<0.1	<0.1
Diethylphthalate	84-66-2	<0.01	<0.01	<0.1	<0.1
Dimethylphthalate	131-11-3	<0.01	<0.01	<0.1	<0.1
Di-n-butylphthalate	84-74-2	<0.01	<0.01	<0.1	<0.1
Di-n-octylphthalate	117-84-0	<0.01	<0.01	<0.1	<0.1
Dinoseb	88-85-7	<0.01	<0.01	<0.1	<0.1
Fluoranthene	206-44-0	<0.01	<0.01	<0.1	<0.1
Fluorene	86-73-7	**0.003J**	**0.0036J**	<0.1	<0.1
Hexachlorobenzene	118-74-1	**0.0046J**	**0.0059J**	**0.053J**	<0.1
Hexachlorobutadiene	87-68-3	<0.01	<0.01	<0.1	<0.1
Hexachlorocyclopentadiene	77-47-4	<0.01	<0.01	<0.1	<0.1
Hexachloroethane	67-72-1	<0.01	<0.01	<0.1	<0.1
Indeno(1,2,3-cd)pyrene	193-39-5	<0.01	<0.01	<0.1	<0.1
Isophorone	78-59-1	<0.01	<0.01	<0.1	<0.1
Naphthalene	91-20-3	**0.02**	**0.021**	<0.1	**0.015J**
Nitrobenzene	98-95-3	**0.0084J**	**0.011**	<0.1	<0.1
N-Nitroso-di-n-propylamine	621-64-7	<0.01	<0.01	<0.1	<0.1
N-Nitrosodiphenylamine	86-30-6	**0.003J**	**0.0027J**	<0.1	<0.1
Pentachlorophenol	87-86-5	**0.15**	**0.16**	**0.13J**	**0.088J**
Phenanthrene	85-01-8	<0.01	<0.01	<0.1	<0.1
Phenol	108-95-2	**0.0054J**	**0.0059J**	<0.1	<0.1
Pyrene	129-00-0	<0.01	<0.01	<0.1	<0.1
Total SVOCs		**2.9**	**3.1**	**2.5**	**1.8**

Notes:
1. All samples were analyzed by Severn Trent Laboratories (STL), Savannah, Georgia.
2. Detected analytes are presented in bold type.
3. J = Analyted detected below quantitation limits. < = Analyte not detected at the specified reporting limit.

3. TEST 2—DISSOLUTION

3.1 Treatability Study Objectives:

Dissolution of contaminants into the aqueous phase was considered as a baseline condition for remediation of the DNAPL source area. No enhancement is achieved, but assessments of both the persistence of contamination and time scales required for clean-up can be generated. This provides a comparison case for natural attenuation and pump-and-treat remediation strategies.

Objectives of the treatability study included:

- Determine whether passive dissolution results in depletion of contaminant mass such that measurable changes in concentration are observed
- Provide design data for pore volumes required to reach specific clean-up levels for the target contaminants and correlate to cost per volume of soil treated

3.2 Technical Approach

Dissolution of DNAPL contamination in the saturated zone of a subsurface region is a function of many factors, including the composition of the DNAPL and the solubility of each individual component (Pankow and Cherry, 1996). These factors place limits on the aqueous phase concentrations that will be present in the vicinity of a source zone. The constant inflow of fresh water from up gradient of the source zone results in steady dissolution into the aqueous phase, leading to the formation of a contaminant plume. As the non-aqueous phase reaches a state of equilibrium with the surrounding matrix, the dissolution rate (mass of contaminant per unit time) becomes relatively constant over time. The rate does gradually decrease as the source zone becomes depleted, and rates at the micro-scale can change more rapidly if DNAPL is widely distributed as ganglia and fingers rather than as pools. However, most DNAPL mass is not particularly mobile once it has been released and allowed to come to equilibrium with the soil and water phases (Pankow and Cherry, 1996). Furthermore, DNAPL that is trapped in the residual state is difficult to recover strictly by pumping water (Wiedemeier et al., 1999). This arises from the challenges in overcoming capillary forces that dominate in the interstitial spaces and hold DNAPL in place.

Because of this, attempts at altering the dissolution rate by manipulating hydraulic factors within an aquifer only minimally impact the source longevity. Rather, it is the initial source mass that appears to be the key factor in determining the amount of time required to completely dissolve a DNAPL source (Wiedemeier et al., 1999). For this reason, treatability tests using flow rates higher than those encountered in the field can provide an indication of dissolution rates without adversely biasing the results. Bench-scale data that provides reasonable estimates of time-to-clean can be generated using short monitoring periods. This data can also be expressed in terms of the number of pore volumes required to reach specific clean-up levels, or a pseudo-dissolution metric where pore volumes replaces time in the denominator.

The COCs detected in recovered DNAPL from the Sauget Area 1 site include 1,2,4-trichlorobenzene, chlorobenzene, 1,2- and 1-4-dichlorobenzene, and benzene. As noted previously, the DNAPL characterization demonstrated that a portion of the DNAPL is composed of a series of unidentified constituents. This unidentified fraction, along with TOC in the soil that is not classified as VOC or SVOC, represents additional compounds that must dissolve into the aqueous phase. Therefore, the dissolution rates for the identified constituents are partially controlled by the mole fraction of each within the DNAPL mixture. This impact of multiple

components on the effective solubility of individual compounds follows the solubility analog of Raoult's Law (Pankow and Cherry, 1996).

Site soil was added to bench-scale columns to model flow-through conditions in an aquifer. Dissolution was quantified in terms of the mass of constituents recovered per pore volume pumped through the column, or the number of pore volumes required to reach clean-up goals. Soil from three distinct depth intervals was used.

3.3 Experimental Methods and Materials

Methods. The test involved monitoring dissolution of contaminants over time in three different soil conditions. Therefore, 3 columns were set-up and analyzed for VOCs and selected SVOCs. Soil was selected from the Area 1 cores that had been used as part of the DNAPL characterization study. Because only a small mass of soil was needed to pack the columns, soil that had been stored at Severn Trent Laboratories (STL) was used for this test. These samples had been sent to STL for VOC/SVOC analysis in October and had been stored in a 4 oz jar at 4°C prior to re-shipping. The soil used for the dissolution tests was taken from samples collected during drilling of piezometers A1-8 at Site I and A1-14 at Site G. The samples selected for testing included soil from 22.5-25 ft and 70-72.5 ft bgs at boring A1-8 and soil from 25-27.5 ft bgs at boring A1-14. These samples were selected based on the presence of elevated levels of VOCs and/or SVOCs in all three intervals, as evidenced by analytical results from the testing conducted by STL.

Column ID	Soil Boring Location (Depth Interval)	Analytes	Sampling Events
C1	A1-08 (22.5-25 ft)	VOC, 1,2-dichlorobenze, 1,3-dichlorobenze, 1,4-dichlorobenzene, 1,2,4-trichlorobenzene	5 PV, 10 PV, 15 PV, 25 PV, 50 PV
C2	A1-08 (70-72.5 ft)	VOC, 1,2-dichlorobenze, 1,3-dichlorobenze, 1,4-dichlorobenzene, 1,2,4-trichlorobenzene	5 PV, 10 PV, 15 PV, 25 PV, 50 PV
C3	A1-14 (25-27.5 ft)	VOC, 1,2-dichlorobenze, 1,3-dichlorobenze, 1,4-dichlorobenzene, 1,2,4-trichlorobenzene	5 PV, 10 PV, 15 PV, 25 PV, 50 PV

The configuration of the flow-through systems is displayed in Figure 3.1. They were constructed from pre-fabricated glass columns with an interior diameter of 1.45 cm and a total length of 16.3 cm. Using a porosity of 0.38 from field data, this yielded an interior pore volume of 10 mL. These columns contained plastic threaded sleeves at both ends that could be connected to metal caps. During packing, only one end of each column was capped while soil was added to the open end. Soil was added through a funnel designed to exclude rocks with a diameter of greater than 4 mm. Columns were lightly tapped throughout the packing process to consolidate sediments and

minimize the formation of air and water pockets. Excess water brought to the top of the columns during this tapping process was transferred out via pipette to ensure that the entire length of the column was filled with sediment.

Following packing, the columns were capped. Each cap was equipped with Swagelok fittings that were connected to 1/8 in. metal lines. The influent line was connected to a syringe pump and valve combination, while the effluent was connected to a 20 mL sealed sampling port. Glass syringes (100 mL) were placed on the pump and used to deliver phosphate-buffered deionized water (pH = 7.2) through the column at a rate of 50 mL/hr. Flow traveled through columns at a Darcy velocity of 7.2 m/d (seepage velocity of 19.1 m/d), corresponding to a residence time of roughly 0.20 hours. After exiting the column, flow was directed through the sampling containers and then to beakers for ultimate disposal. Teflon-coated tape was used on all sleeves to prevent leaking, and small circular mesh screens were cut and placed at both the influent and effluent ends of the packed media to prevent soil from leaving the column and becoming caught in the lines.

Samples were taken at 5 time intervals that represented 5, 10, 15, 25 and 50 pore volumes of water. At the flow rate used, this corresponded to a 10 hr sampling period. The sampling containers for each column were sealed and therefore suitable for measuring VOCs. Because the syringe pump contained slots for only two syringes, it was necessary to separate the experimental monitoring into two different sampling periods. Columns C1 and C2 were run simultaneously on December 3, while column C3 was run on December 6.

Analysis. Samples for analysis of volatile organic compounds (VOCs) were collected in 40 mL vials with TFE. Vials were filled with no headspace, and hydrochloric acid was used as a preservative. Samples were collected from the sealed sampling devices at the effluent end of the column via disposable syringes and 4 in. long 20 gauge needles. This volume (17 to 19 mL) was transferred to the vials and diluted with an equal amount of deionized water to reach a total volume of 40 mL. Samples were collected and analyzed using a modified version of EPA Method 8260. This method used a longer analytical run that allowed for identification and quantification of selected SVOCs, specifically 1,2,4-trichlorobenzene, 1,2-dichlorobenzene, and 1,4-dichlorobenzene. All sample analyses were conducted by Severn Trent Laboratories (STL). Collected samples were shipped overnight in coolers on ice, and all bottles were stored at 4ºC until ready to ship.

3.4 Results and Discussion

The data collected for VOC concentrations versus pore volumes for the three columns are located in Table 3.1. This data was then corrected for sample dilution to yield the concentrations listed in Table 3.2. A number of constituents were detected in the effluent of all three columns, and concentration trends were assessed based on grouping of these constituents as volatile or semi-volatile. The total COC concentration per pore volume passing through each column was also monitored.

As shown in Figure 3.2(a) through Figure 3.4(a), total COC concentrations near the start of the pumping cycle (5 PV) were measured as 11,070 μg/L from C1, 26,568 μg/L from C2, and 3768 μg/L from C3. Over the course of 50 pore volumes, passive dissolution resulted in partial depletion of contaminant mass, but only minimal changes in total COC concentration were observed, suggesting that dissolution rates reached steady-state within a short period following the initiation of pumping. After 50 pore volumes, the total COC concentrations had decreased but within a range of only 10 to 30%. This observation that COC concentrations remained relatively level over 50 pore volumes suggests that a large portion of the contaminant profile is characterized by non-aqueous compounds that are highly subject to partitioning and retardation

effects. This is not surprising given that distillation tests with recovered DNAPL have determined a large percentage of the contaminants (at least 75% by volume) contain more than six carbons. As a group, longer chain hydrocarbons and aromatics tend to have higher organic-water partitioning coefficients than shorter-chain aliphatics and simple aromatics.

The flat concentration profile over time is also consistent with studies indicate that concentration changes in soil matrices containing NAPL are dependent on mass removal, and that significant changes in concentration are generally preceded by large changes in source mass (Newell and Adamson, 2004; Sale and McWhorter, 2001). In the case of the three soil samples used to create these columns, the total mass of COC present initially can be estimated using previous analyses of the cores by STL. These calculations are detailed in Table 3.3. Using C1 (soil boring A1-08 22.5-25' bgs) as an example, 48 g of soil containing 25420 mg/kg of COCs was used, yielding a total COC mass of 1220 mg in the column. Over the course of 50 pore volumes (500 mL), a total of 4.6 mg of COCs were pumped out of the column, or 0.38%. Higher removal percentages were observed in both C2 (35%) and C3 (3.0%), but the data suggests that more significant declines in the total COC concentration would not be expected until mass removal was more substantial.

While dissolution rates for COCs are a function of the number of pore volumes that have been pumped through each column, the rates did not change substantially over the course of the monitoring period. This is noted by the cumulative mass curves in Figures 3.2(b) through 3.4(b). The dissolution rate for each column is essentially the slope of these curves (after converting the pore volumes to time-based units using the flow rate), and the relative straightness of each demonstrates consistent rates. The dissolution rate after 50 pore volumes ranged from 0.16 mg/hr for column C3, 0.46 mg/hr for column C1, and 1.28 mg/hr for column C2. Assuming that the dissolution rates after 50 pore volumes are equal to the value at 50 pore volumes, the number of pore volumes required to deplete the remaining DNAPL mass can be estimated. This is equivalent to assuming that the dissolution rate follows a step function model (Sale and McWhorter, 2001). Using this approach, **it would require 13,300 pore volumes to deplete the remaining mass of DNAPL in C1, 144 pore volumes to deplete the remaining DNAPL in C2, and 1,650 pore volumes to deplete the remaining mass of DNAPL in C3.**

Alternatively, the concentration and pore volume data can be modeled based on a first-order decay relationship. This is displayed in Figure 3.2(c) through Figure 3.4(c) for the concentrations observed from 10 to 50 pore volumes. Because the concentration at the effluent represents time-course data for a single point that is representative of the entire contaminant volume, it can be used to estimate duration of the plume. In the case of the Sauget Area 1 site, the time required to decrease the total concentration by three orders of magnitude ($C/C_o = 0.001$) is a potential goal (e.g., this concentration reduction would reduce most of the constituents below Illinois Class I standards for groundwater). This yields a target effluent concentration ranging from 1 ug/L for column C1, 27 ug/L for column C2, and 3.8 ug/L for column C3. This is equivalent to effluent concentrations that are lower than the individual . The first order decay coefficient (k_s) for each column is generated by calculating the slope after plotting concentration data on a log scale. The pore volumes required can then be generated using the following transformed first-order relationship:

$$t = - \ln (C_{goal}/C_{start})/k_s \quad \text{(where t is in terms of pore volumes)}$$

This yields a value of **743 pore volumes required to reach the goal concentration for column C1** (soil from A1-08 22.5-25' interval) and **2763 pore volumes required to reach the goal concentration for column C2** (soil from A1-08 70-72.5' interval). For column C3, the k_s value is positive (0.0021/pore volume), meaning that the concentration trend over the course of 50 pore

volumes was not downward and a sufficient concentration projection over a longer period was not possible.

The above analyses rely on bulk dissolution of the entire suite of constituents detected in the effluent. A similar analysis could be performed for each of the individual constituents, although this type of dissolution analysis can be compromised by a constantly changing DNAPL composition. Specifically, the effective solubility of each compound is controlled by its relative mole fraction, and the rapid dissolution of a more soluble compound will impact the subsequent dissolution patterns of those less soluble compounds as they become a primary component of the remaining DNAPL mixture. Therefore, the dissolution values generated in this treatability test should not be extended beyond bulk estimates. Despite this limitation, concentration patterns of individual constituents in each column are worth noting. For example, in column C2, 1,4-dichlorobenzene comprised the majority of the contaminant mass in the effluent. Aqueous concentrations of 1,4-dichlorobenzene remained high throughout the monitoring period, but a number of other compounds that were initially present decreased. Of the COCs monitored in column C2, chlorobenzene, tetrachloroethene, and 1,2-dichlorobenzene decreased below their Illinois Class I groundwater standards by the conclusion of the monitoring period. In column C1, the effluent concentration was dominated by the chlorinated benzenes, all of which remained at high levels throughout the course of the monitoring period. However, several VOCs decreased to levels below the Class I standards, specifically methylene chloride, acetone, MEK, and MIBK. Similarly, the effluent concentration in column C3 was comprised of mainly SVOCs (specifically 1,2,4-trichlorobenzene), and these concentrations remained relatively constant after 50 pore volumes. Only MIBK and benzene decrease to levels below the Class I standards.

As expected, dissolution in the columns resulted in more significant decreases in concentration for those compounds that are more water-soluble and less sorptive (e.g. methylene chloride, benzene, MIBK, MEK), and concentrations of less soluble compounds (notably 1,2,4-trichlorobenzene) did not change. The compounds detected in the effluents of each soil column matched the contaminant profile generated by the previous analysis of the soil.

Translating this data to the Sauget Area 1 site requires some estimate of the mass (or volume) of soil required to remediate, as well as the approximate groundwater velocity (or flow rate). This can be accomplished by estimating the time for a pore volume to pass through a given volume of soil. The bench-scale treatability test is not intended to provide an exact value for the time required to deplete the contaminant mass, and certain constituents could be depleted faster than the bulk estimates generated from the entire COC mass.

GROUNDWATER SERVICES, INC.

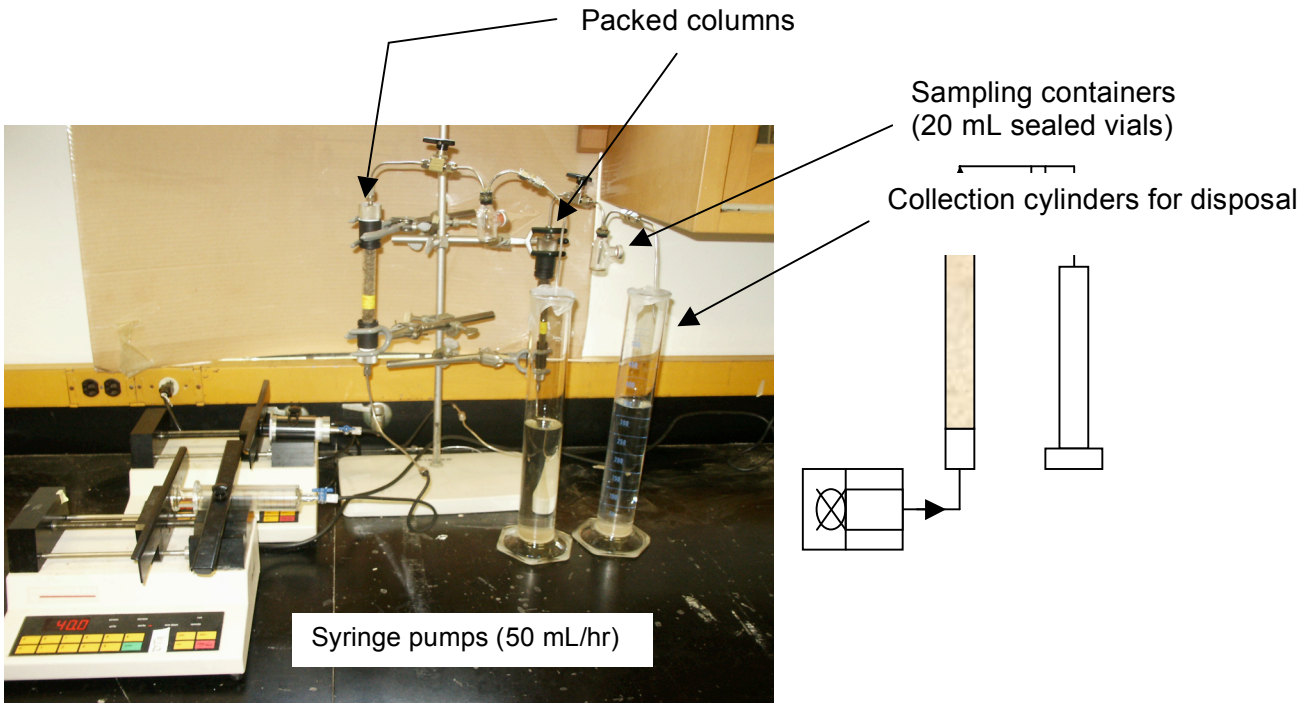
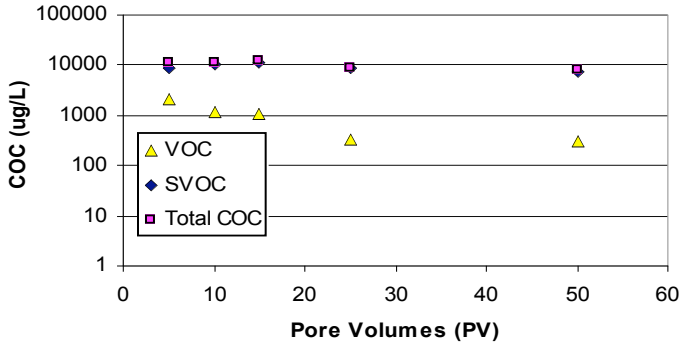
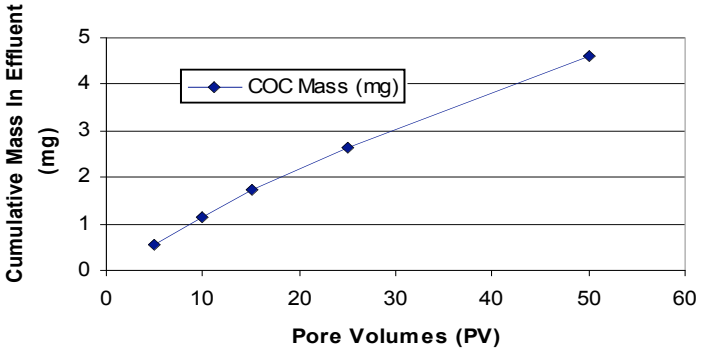


Figure 3.1. Flow-through columns used for Dissolution Treatability Tests.

GROUNDWATER SERVICES, INC.



(a)



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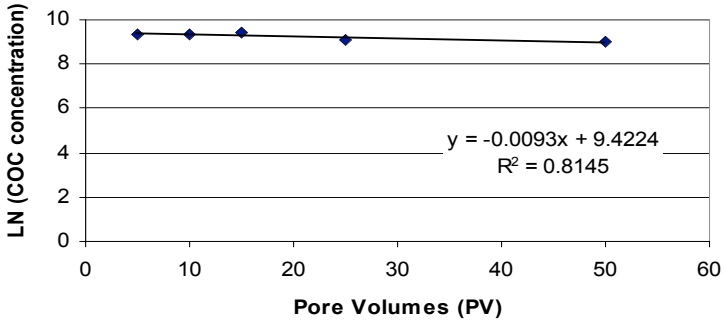
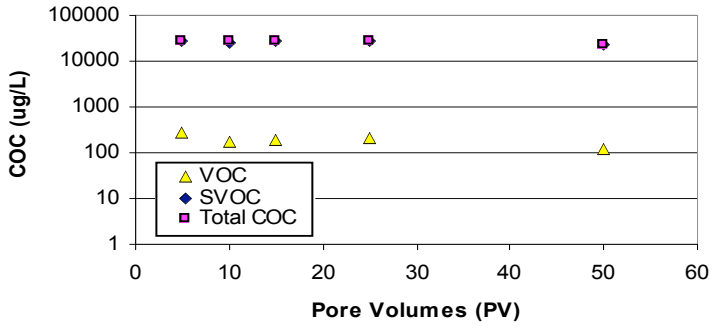


(c)

Figure 3.2. Dissolution treatability test for Column C1 (soil from A1-08 22.5-25' bgs) (a) Concentration of VOCs, SVOCs, and COCs for pore volumes pumped through column, (b) Cumulative mass pumped through column, and (c) Concentration of VOCs (mg/L on a natural log scale) for pore volumes. The slope of the line in Figure 3.2(c) represents the first order decay coefficient ($-k_s$) in units of (pore volume)$^{-1}$.

GROUNDWATER SERVICES, INC.



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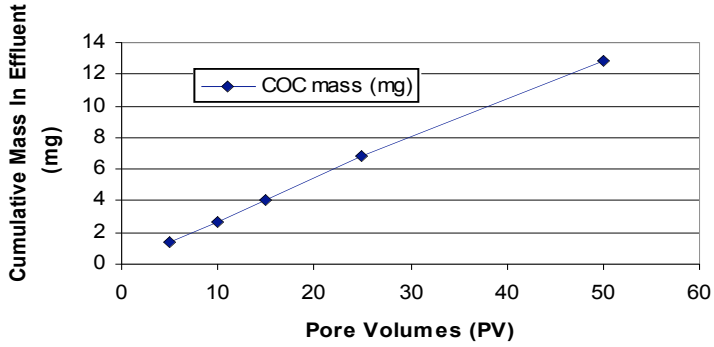
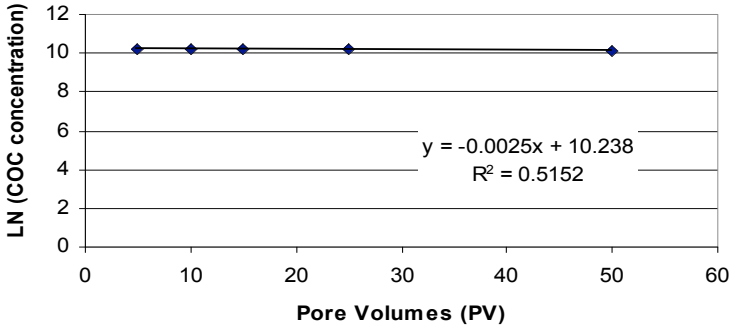


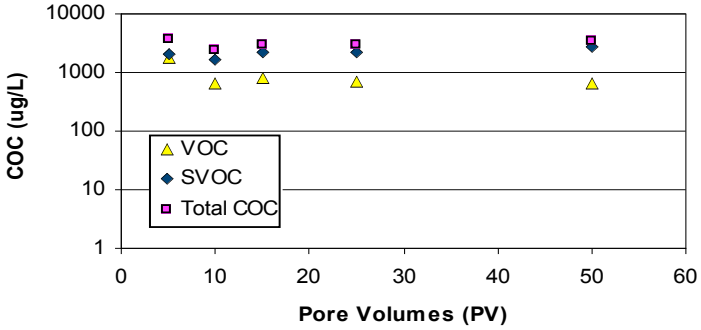
(b)



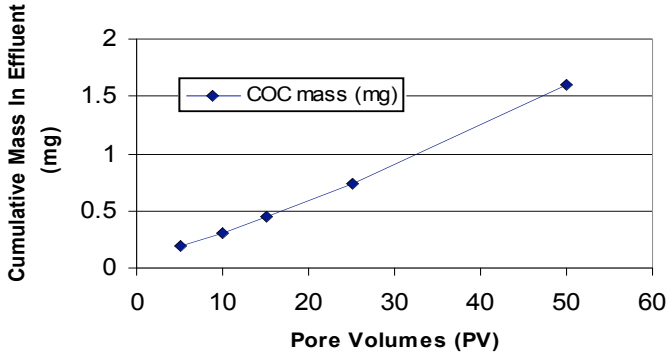
(c)

Figure 3.3. Dissolution treatability test for Column C2 (soil from A1-08 70-72.5' bgs) (a) Concentration of VOCs, SVOCs, and COCs for pore volumes pumped through column, (b) Cumulative mass pumped through column, and (c) Concentration of VOCs (mg/L on a natural log scale) for pore volumes. The slope of the line in Figure 3.3(c) represents the first order decay coefficient ($-k_s$) in units of (pore volume)$^{-1}$.

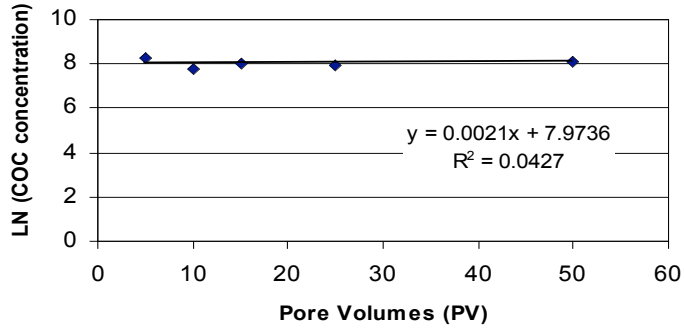
GROUNDWATER SERVICES, INC.



(a)



(b)



(c)

Figure 3.4. Dissolution treatability test for Column C3 (soil from A1-14 25-27.5' bgs) (a) Concentration of VOCs, SVOCs, and COCs for pore volumes pumped through column, (b) Cumulative mass pumped through column, and (c) Concentration of VOCs (mg/L on a natural log scale) for pore volumes. The slope of the line in Figure 3.4(c) represents the first order decay coefficient ($-k_s$) in units of $(\text{pore volume})^{-1}$.

Table 3.1
Total VOC Concentrations: Dissolution Treatability Test
(Diluted Samples)

DNAPL Treatability Study
Sauget Area 1, Sauget, Illinois

	SAMPLE ID:	C1-5 PV	C1-10 PV	C1-15 PV	C1-25 PV	C1-50 PV	C2-5 PV	C2-10 PV	C2-15 PV	C2-25 PV	C2-50 PV	C3-5 PV	C3-10 PV	C3-15 PV	C3-25 PV	C3-50 PV
	SAMPLE DATE:	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/6/04	12/6/04	12/6/04	12/2/04	12/2/04
Analyte	**CAS No.**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**
Volatile and Semi-Volatile Organic Compounds by EPA Method 8260																
1,1,1-Trichloroethane	71-55-6	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
1,1,2,2-Tetrachloroethane	79-34-5	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
1,1,2-Trichloroethane	79-00-5	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
1,1-Dichloroethane	75-34-3	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
1,1-Dichloroethene	75-35-4	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
1,2,4-Trichlorobenzene	120-82-1	**2.5E**	**2.9E**	**3.1E**	**2.5E**	**1.9E**	**0.3**	**0.14**	**0.13**	**0.14**	**0.074**	**.7E**	**.59E**	**.75E**	**0.76E**	**1E**
1,2-Dichlorobenzene	95-50-1	**0.5**	**0.53**	**0.58**	**0.42**	**0.44**	**0.32**	**0.28**	<0.005	<0.005	**0.22**	**0.027**	**0.021**	**0.022**	**0.022**	**0.023**
1,2-Dichloroethane	107-06-2	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
1,2-Dichloropropane	78-87-5	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
1,3-Dichlorobenzene	541-73-1	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
1,4-Dichlorobenzene	106-46-7	**0.85**	**0.96**	**1E**	**0.79**	**0.86**	**1.2E**	**1.2E**	**1.3E**	**1.3E**	**1.1E**	**0.14**	**0.11**	**0.14**	**0.14**	**0.14**
2-Butanone (MEK)	78-93-3	**0.012J**	**0.0086J**	**0.0098J**	<0.05	<0.05	**0.0065J**	<0.05	<0.05	**0.0068J**	**0.009J**	<0.02	**0.0032J**	<0.02	**0.0041J**	**0.0083J**
2-Hexanone	591-78-6	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	**0.0022J**	<0.02	<0.02	<0.02	<0.02
4-Methyl-2-pentanone (MIBK)	108-10-1	**0.058**	**0.028J**	**0.022J**	**0.0034J**	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	**0.38**	**0.022**	**0.0035J**	<0.02	<0.02
Acetone	67-64-1	**0.59**	**0.28**	**0.24**	<0.12	<0.12	**0.036J**	<0.12	<0.12	<0.12	<0.12	**0.014J**	<0.05	<0.05	**0.01J**	<0.05
Benzene	71-43-2	**0.02**	**0.016**	**0.015**	**0.0091**	**0.0086**	<0.005	<0.005	<0.005	<0.005	<0.005	**0.045**	**0.037**	**0.044**	**0.022**	**0.0022**
Bromodichloromethane	75-27-4	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
Bromoform	75-25-2	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
Bromomethane	74-83-9	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
Carbon Disulfide	75-15-0	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
Carbon Tetrachloride	56-23-5	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
Chlorobenzene	108-90-7	**0.13**	**0.12**	**0.13**	**0.091**	**0.093**	**0.062**	**0.056**	**0.066**	**0.067**	**0.031**	**0.032**	**0.026**	**0.035**	**0.033**	**0.036**
Chloroethane (ethyl chloride)	75-00-3	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
Chloroform	67-66-3	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	**0.0011J**	<0.002	<0.002	<0.002	<0.002
Chloromethane	74-87-3	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
cis-1,2-Dichloroethene	156-59-2	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	**0.0037**	**0.0014J**	<0.002	<0.002	<0.002
cis-1,3-Dichloropropene	10061-01-5	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
Dibromochloromethane	124-48-1	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
Dichloromethane	75-09-2	**0.004J**	**0.0042J**	**0.0047J**	**0.0036J**	**0.0037J**	**0.016**	**0.015**	**0.018**	**0.019**	**0.015**	**0.062**	**0.048**	**0.066**	**0.064**	**0.067**
Ethyl benzene	100-41-4	**0.0037J**	**0.0037J**	**0.0034J**	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	**0.011**	**0.0015J**	<0.01	<0.01	<0.01
Styrene	100-42-5	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
Tetrachloroethene	127-18-4	**0.011**	**0.0086**	**0.0084**	**0.0081**	**0.0046J**	**0.0083**	**0.0093**	**0.0049J**	**0.0044J**	**0.002J**	**0.012**	**0.0065**	**0.0052**	**0.0036**	**0.003**
Toluene	108-88-3	**0.016**	**0.016**	**0.016**	**0.011**	**0.012**	<0.005	<0.005	<0.005	<0.005	<0.005	**0.16**	**0.13**	**0.17**	**0.15**	**0.16**
trans-1,2-Dichloroethene	156-60-5	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
trans-1,3-Dichloropropene	10061-02-6	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
Trichloroethene	79-01-6	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	**0.0066**	**0.0049**	**0.0066**	**0.0066**	**0.0034**
Vinyl Chloride	75-01-4	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
Xylenes,Total	1330-20-7	**0.01**	**0.011**	**0.012**	**0.0092J**	**0.01**	<0.01	<0.01	<0.01	<0.01	<0.01	**0.005**	**0.0041**	**0.0053**	**0.0051**	**0.0056**
Total VOCs + SVOCs		**4.7**	**4.9**	**5.1**	**3.8**	**3.3**	**13**	**13**	**13**	**13**	**11**	**1.6**	**2.0**	**1.2**	**1.2**	**1.4**

Notes:
1. All samples were analyzed by Severn Trent Laboratories (STL), Savannah, Georgia.
2. Detected analytes are presented in bold type.
3. J = Analyted detected below quantitation limits. < = Analyte not detected at the specified reporting limit. E = Result exceeds calibration range.

Table 3.2
Total VOC Concentrations: Dissolution Treatability Test
(Results Corrected for Sample Dilution)

DNAPL Treatability Study
Sauget Area 1, Sauget, Illinois

	SAMPLE ID:	C1-5 PV	C1-10 PV	C1-15 PV	C1-25 PV	C1-50 PV	C2-5 PV	C2-10 PV	C2-15 PV	C2-25 PV	C2-50 PV	C3-5 PV	C3-10 PV	C3-15 PV	C3-25 PV	C3-50 PV
	SAMPLE DATE:	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	12/6/04	12/6/04	12/6/04	12/2/04	12/2/04
Analyte	**CAS No.**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**	**mg/l**
Volatile and Semi-Volatile Organic Compounds by EPA Method 8260																
1,2,4-Trichlorobenzene	120-82-1	**5.88**	**6.82**	**7.29**	**5.88**	**4.47**	**0.63**	**0.29**	**0.27**	**0.29**	**0.16**	**1.65**	**1.39**	**1.76**	**1.79**	**2.35**
1,2-Dichlorobenzene	95-50-1	**1.18**	**1.25**	**1.36**	**0.99**	**1.04**	**0.67**	**0.59**			**0.46**	**0.064**	**0.049**	**0.052**	**0.052**	**0.054**
1,4-Dichlorobenzene	106-46-7	**2.00**	**2.26**	**2.35**	**1.86**	**2.02**	**2.53**	**2.53**	**2.74**	**2.74**	**2.32**	**0.33**	**0.26**	**0.33**	**0.33**	**0.33**
2-Butanone (MEK)	78-93-3	**0.028**	**0.020**	**0.023**			**0.014**			**0.014**	**0.019**		**0.008**		**0.010**	**0.020**
2-Hexanone	591-78-6											**0.005**				
4-Methyl-2-pentanone (MIBK)	108-10-1	**0.14**	**0.066**	**0.052**	**0.008**							**0.89**	**0.052**	**0.008**		
Acetone	67-64-1	**1.39**	**0.66**	**0.56**			**0.076**					**0.033**			**0.024**	
Benzene	71-43-2	**0.047**	**0.038**	**0.035**	**0.021**	**0.020**						**0.11**	**0.087**	**0.10**	**0.052**	**0.005**
Chlorobenzene	108-90-7	**0.31**	**0.28**	**0.31**	**0.21**	**0.22**	**0.13**	**0.12**	**0.14**	**0.14**	**0.065**	**0.075**	**0.061**	**0.082**	**0.078**	**0.085**
Chloroform	67-66-3											**0.003**				
cis-1,2-Dichloroethene	156-59-2											**0.009**	**0.003**			
Dichloromethane	75-09-2	**0.009**	**0.010**	**0.011**	**0.008**	**0.009**	**0.034**	**0.032**	**0.038**	**0.040**	**0.032**	**0.15**	**0.11**	**0.16**	**0.15**	**0.16**
Ethyl benzene	100-41-4	**0.009**	**0.009**	**0.008**								**0.03**	**0.00**			
Tetrachloroethene	127-18-4	**0.026**	**0.020**	**0.020**	**0.019**	**0.011**	**0.017**	**0.020**	**0.010**	**0.009**	**0.004**	**0.028**	**0.015**	**0.012**	**0.008**	**0.007**
Toluene	108-88-3	**0.038**	**0.038**	**0.038**	**0.026**	**0.028**						**0.38**	**0.31**	**0.40**	**0.35**	**0.38**
Trichloroethene	79-01-6											**0.016**	**0.012**	**0.016**	**0.016**	**0.008**
Xylenes,Total	1330-20-7	**0.024**	**0.026**	**0.028**	**0.022**	**0.024**						**0.012**	**0.010**	**0.012**	**0.012**	**0.013**
Total VOCs + SVOCs		**11.1**	**11.5**	**12.1**	**9.0**	**7.8**	**4.1**	**3.6**	**3.2**	**3.2**	**3.1**	**3.8**	**2.4**	**2.9**	**2.9**	**3.4**

Notes:
1. All samples were analyzed by Severn Trent Laboratories (STL), Savannah, Georgia.
2. Detected analytes are presented in bold type.
3. Concentrations displayed were calculated by converting the concentrations listed in Table 3.1 using the appropriate dilution factor.

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Table 3.3
Mass Calculations
Dissolution Treatability Test
Sauget Area 1, Sauget, Illinois

Sample ID		C1-5PV	C1-10PV	C1-15PV	C1-25PV	C1-50PV	
Date Sampled		12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	
TOTAL VOC	(µg/L)	2011	1167	1085	319	310	
TOTAL SVOC	(µg/L)	9059	10329	11012	8729	7529	
TOTAL COC	(µg/L)	11070	11497	12097	9048	7840	
Pore Volumes (PV)		5	10	15	25	50	
Cumulative mass in effluent (mg)		**0.55**	**1.13**	**1.73**	**2.64**	**4.60**	
Original Mass in Column (at onset of pumping) (mg)							**1220**
% of Original Mass Recovered in Effluent after 50 Pore Volumes							**0.38%**

Sample ID		C2-5PV	C2-10PV	C2-15PV	C2-25PV	C2-50PV	
Date Sampled		12/2/04	12/2/04	12/2/04	12/2/04	12/2/04	
TOTAL VOC	(µg/L)	2011	1167	1085	319	310	
TOTAL SVOC	(µg/L)	26568	26147	27642	27663	23777	
TOTAL COC	(µg/L)	26840	26316	27829	27868	23897	
Pore Volumes (PV)		5	10	15	25	50	
Cumulative mass in effluent (mg)		**1.34**	**2.66**	**4.05**	**6.84**	**12.81**	
Original Mass in Column (at onset of pumping) (mg)							**36.8**
% of Original Mass Recovered in Effluent after 50 Pore Volumes							**34.80%**

Sample ID		C3-5PV	C3-10PV	C3-15PV	C3-25PV	C3-50PV	
Date Sampled		12/6/04	12/6/04	12/6/04	12/6/04	12/6/04	
TOTAL VOC	(µg/L)	271	169	187	205	120	
TOTAL SVOC	(µg/L)	2040	1696	2146	2169	2736	
TOTAL COC	(µg/L)	3768	2366	2936	2872	3408	
Pore Volumes (PV)		5	10	15	25	50	
Cumulative mass in effluent (mg)		**0.19**	**0.31**	**0.45**	**0.74**	**1.59**	
Original Mass in Column (at onset of pumping) (mg)							52.5
% of Original Mass Recovered in Effluent after 50 Pore Volumes							3.04%

4. THERMAL TREATABILITY EVALUTION

4.1 Introduction

Thermal treatment is a general term for a variety of approaches designed to destroy or mobilize constituent mass *in situ*. Most methods involve the injection of heat (often in the form of steam) to vaporize and strip volatile compounds. In such cases, vacuum wells are necessary to capture and recover the vapor phase constituents. If higher temperatures are employed, constituents can be completely oxidized or pyrolyzed.

4.2 Technical Approach

Injection wells deliver heat or steam from the surface to the contaminated zone. A portion of a steam input vaporizes NAPL and continues to rise above the treatment zone, while condensed steam raises bulk soil temperatures to above the boiling points of many dissolved volatile constituents (enhancing vaporization and solubilization). Alternatively, steam can be generated *in situ* by thermal conduction from the surface. Soil/vapor extraction wells are placed to strategically capture all volatilized compounds, either as a separate series of wells or via injection wells that serve a dual role as vacuum wells. Higher temperature applications can use thermal conduction to completely boil off all water within the treatment zone, followed by further heating (often > 700°C) to desorb and volatilize semi-volatile compounds. The temperature of the vacuum/extraction wells can often be hot enough to promote oxidation/pyrolization (US DOE, 2000).

The largest application of this technology has been the Visalia site in California, a combination of Dynamic Underground Stripping and Hydrous Pyrolysis Oxidation (DUS/HPO), to treat PCP, creosote, and diesel (US DOE, 2000). Thermal treatment was applied over a ~4 acre, ~600,000 cubic yards treatment zone. This compares to a 15 acre, 1.7 million cubic yard treatment zone for Sauget Area 1 (i.e., the zone containing DNAPL).

At the Visalia site, steam and oxygen were continuously injected to heat the aquifer to the boiling point of water and mobilize a portion of the contamination through volatilization and stripping. This portion was captured in extraction wells, though it was not necessary to boil off all of the residual water. At a specific interval, steam injection was shut off, and condensing steam reacted with remaining constituents (specifically low vapor pressure components such as PCP) and oxygen to promote oxidation at favorably high temperatures. The high temperatures also stimulated biological degradation of the constituents, and hydraulic control was responsible for recovery of a portion of the overall mass. Therefore, the DUS/HPO system was part of an integrated approach to remediating the aquifer (Figure 4.1). During the twenty-five months of operation, approximately 50% of the contaminants were removed in the free phase, 16% as hydrocarbon vapors, 16% in the aqueous phase, and 17% were destroyed by hydrous pyrolysis in situ.

4.3 Design Data

The principal constituents by mass fraction in the DNAPL sample from well BR-I were 1,2,4-trichlobenzene (14%); hexachlorobenzene (1%); and 1,4-dichlorobenzene (0.8%). These chemicals have minimum boiling points of 416°F, 630°F, and 346°F, respectively. Distillation test results using recovered DNAPL from well BR-I indicate that only 5% of the DNAPL has a boiling point at or below 432°F (see laboratory report in Section C.2 of Appendix C). The remaining 83%

of the sample volume recovered had a boiling point that fell within the relatively narrow range of 432 and 530°F.

The DNAPL constituents within the fill materials and alluvial aquifer at Sauget Area 1 have relatively high boiling points, which indicates that volatilization is not likely to be the predominant source removal mechanism during thermal treatment using the DUS/HPO technology. Instead the predominant mass removal mechanism is likely to be pumping of free product, based on results from the Visalia site. Heating of the fill materials and aquifer matrix would reduce interfacial tension and viscosity of residual DNAPL, thereby increasing the potential for the DNAPL to move through the fill and aquifer matrix and be removed by pumping from recovery wells.

The boiling point range for the Sauget Area 1 DNAPL is similar to the boiling point range for the Visalia site, which had creosote type compounds with a minimum boiling point 397°F and PCP with a boiling point of 588°F. Based on the performance of the Visalia system, **significant (but not complete) DNAPL recovery could be expected from a DUS/HPO treatment at Sauget Area 1** (i.e., thermal treatment without dewatering the saturated zone). Estimated costs for the HPO/DUS technology implemented at Visalia range from $75 to $100 per cubic yard of soil remediated.

While significant mass was removed from the Visalia Site, there would be additional technical challenges to applying this technology at Sauget Area 1 compared to Visalia:

1) The size of the application area for Sauget Area 1 would be larger than the application area for the Visalia project;

2) Fifty percent of the creosote removed from the Visalia Site was removed as a mobilized *free phase* through three permeable layers at the site. At Sauget Area 1, there is a single ~100 thick saturated zone. Mobilizing large volumes of DNAPL through this thick treatment zone could be very difficult to control without risk of lateral NAPL migration.

3) The transmissivity of the Sauget Area 1 water-bearing unit is much greater than the transmissivity of the water-bearing units at the Visalia site, and this would increase the scale and cost of the required hydrologic control component of the thermal treatment system.

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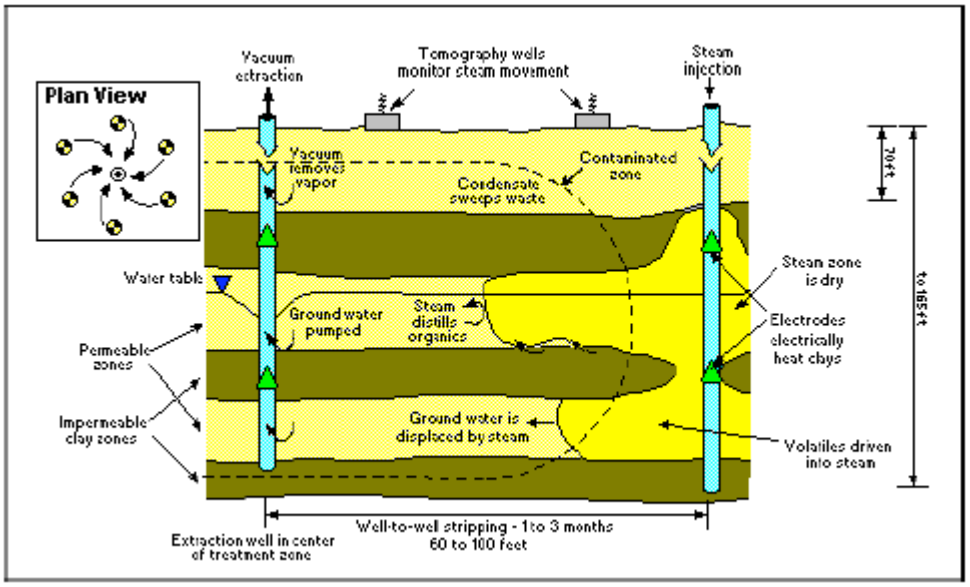

Figure 4.1. Conceptual Model of the DUS/HPO process employed at the Visalia Superfund Site (adapted from US DOE Innovative Technology Summary Report, 2000).

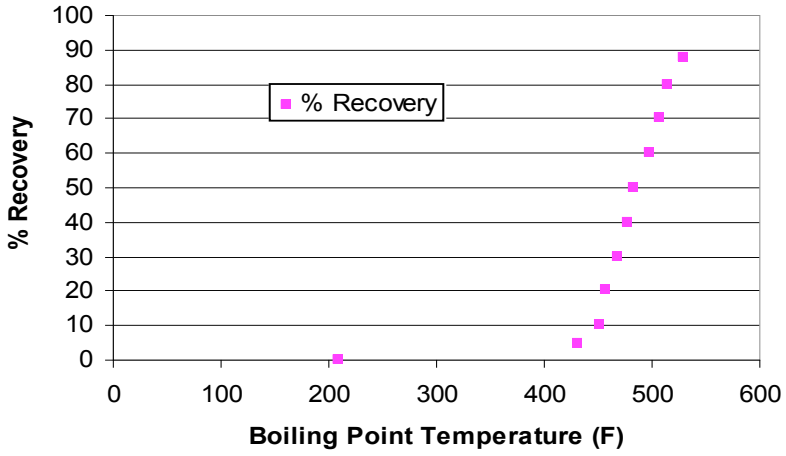

Figure 4.2. Recovery of Sauget Area 1 DNAPL during ASTM-D-86 Distillation Test. Initial boiling point for the DNAPL was 210°F, and 88% of the total volume was recovered at the final boiling point of 530°F.

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5. CHEMICAL OXIDATION TREATABILITY EVALUTION

5.1 Evaluation Objectives:

Chemical oxidation was considered as another potential aid to accelerate remediation of the DNAPL source area. Enhancement is achieved by the chemical reaction of a strong oxidant with a reduced contaminant with the goal of directly converting the compound to CO_2. This chemical attack is direct and can be applied as an *in situ* remedial strategy, thus reducing the costs associated with down gradient treatment and/or excavation.

The objective of the evaluation is:

- Provide design data for mass of oxidant required per mass of DNAPL present and correlate to cost per volume of soil treated

5.2 Technical Approach

Chemical oxidation using potassium permanganate is an extensively studied technology for use in treating both aqueous and non-aqueous phase contaminants. While the selection of oxidant may differ depending on site-specific needs, the goal of any oxidant-based treatment is to promote mass destruction of a reduced contaminant (Yin and Allen, 1999). This occurs through a thermodynamically favorable chemical oxidation in which the contaminant accepts electrons generated from the reduction of the added oxidant. Common chemicals used for this purpose include hydrogen peroxide (H_2O_2), chloride dioxide (ClO_2), and potassium permanganate ($KMnO_4$). The latter has been used for removing drinking water pollutants for several decades, and it has been applied in field demonstrations for removing DNAPL at the Borden site (Schnarr et al., 1998) and at the Portsmouth Gaseous Diffusion Plant in Ohio (U.S. DOE). Potassium permanganate was selected for the evaluation described here because of this previous success in treating DNAPL and because of its ease of use.

In the process of oxidizing contaminants, the permanganate ion is reduced to the solid precipitate manganese dioxide (Yin and Allen, 1999). If sufficient permanganate is present, complete oxidation of the contaminant to CO_2 occurs. The remaining by-products (water, salts) are similarly harmless. While oxidation is typically most efficient for compounds with double bonds, it can be used for single-bonded carbon provided sufficient amounts of the oxidant are supplied. Compounds identified in the Sauget Area 1 DNAPL include chlorobenzene, dichlorobenzene, and trichlorobenzene. The amount of permanganate needed to completely oxidize these compounds can be determined from the following reaction stoichiometries:

Chlorobenzene: $C_6H_5Cl + 9.3\ KMnO_4 + 8.3\ H^+ \rightarrow 6\ CO_2 + 9.3\ MnO_2 + 6.7\ H_2O + Cl^- + 9.3\ K^+$

Dichlorobenzene: $C_6H_4Cl_2 + 8.7\ KMnO_4 + 6.7\ H^+ \rightarrow 6\ CO_2 + 8.7\ MnO_2 + 5.3\ H_2O + 2\ Cl^- + 8.7\ K^+$

Trichlorobenzene: $C_6H_3Cl_3 + 8\ KMnO_4 + 5\ H^+ \rightarrow 6\ CO_2 + 8\ MnO_2 + 4\ H_2O + 3\ Cl^- + 8\ K$

On a mass basis, this corresponds to ratios of **13.1 mg of $KMnO_4$ required per mg of chlorobenzene, 9.3 mg of $KMnO_4$ required per mg of dichlorobenzene, and 7.0 mg of $KMnO_4$ required per mg of trichlorobenzene**.

5.3 Discussion

From an engineering standpoint, these reactions assume complete mineralization of contaminant and do not account for competing side reactions with background organic carbon in the soil. While an insufficient supply of permanganate (or poor contact between oxidant and contaminant) may lead to the formation of incomplete oxidation products, it is likely that the majority would be

cleaved-ring by-products and thus more prone to further attenuation by biotic or abiotic processes. Other compounds, like benzene and MTBE, have not proven particularly susceptible to oxidation by potassium permanganate (US EPA, 2004).

The addition of a chemical oxidant can provide an added benefit by enhancing the dissolution of DNAPL into the surrounding groundwater. Potassium permanganate is active in the aqueous phase, and it can only work as an oxidant on an aqueous phase contaminant. Therefore, the oxidant acts to increase mass flux of the contaminant out of the DNAPL as the chemical reaction depletes aqueous phase contaminant. This decreases the source longevity, which is proportional to the remediation time. This effect, combined with the *in situ* destruction of contaminant, demonstrates that chemical oxidation strategies are less dependent on advective flushing than surfactant addition (Pankow and Cherry, 1996). Chemical oxidation has the potential to be a cheaper technology when down-gradient recirculation schemes are employed to promote complete mixing and contact, as well as to improve hydraulic control of the oxidant and contaminants.

The COCs targeted at the Sauget Area 1 include chlorobenzene, 1,2- and 1-4-dichlorobenzene, and 1,2,4-trichlorobenzene. In addition, the DNAPL characterization and distillation tests demonstrate that a portion of the DNAPL is accounted for by a series of unidentified long-chain hydrocarbons. This unidentified fraction, along with TOC in the soil that is not classified as VOC or SVOC, represents an additional oxidant demand that cannot be calculated using stoichiometry. Potassium permanganate has the potential to catalyze the oxidation of all organic carbon regardless of whether or not is part of the contaminant fraction, but it is non-selective. Furthermore, many compounds in the contaminant profile (such as benzene) may not be readily oxidized.

A previous treatability study using site soil from the Krummich facility at Sauget indicated that potassium permanganate was not successful in converting the entire constituent mass to CO_2. The tests yielded ratios ranging from 15.7 to 148.3 g of permanganate needed per g of VOC oxidized, in part because the oxidation reaction was kinetically limited. Combined with the stoichometrically-derived values, these ratios can be used as conservative estimates of the mass of oxidant needed at Sauget Area 1. The soil data collected at the site provides an estimate of the mass of COCs present, and this mass can be used directly or converted to a unit volume of soil. The addition of oxidant does have the potential to mineralize contaminant mass, but sufficient time and contact are necessary to ensure the oxidation process is complete. At 12 sites where complete oxidation of target contaminants was achieved, a median unit cost of \$67/yd^3 has been reported (McDade et al., 2004), with 25-75% of the sites falling between \$27 and \$196/yd^3. The largest site in the SERDP database for this technology is 25,500 yd^3. The estimate of the remedial volume at Sauget Area 1 is 1.7 million yd^3, which is 72 times the largest volume reported for treatment by chemical oxidation.

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